

FINAL REPORT
ON
WASH WATER WASTE PRETREATMENT SYSTEM

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Contract NAS 9-14965

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SUMMARY

Under Contract NAS 9-14965, investigations were completed on wash waters based on each candidate personal cleansing agent - including evaluations of coagulants, antifoam agents, and the effect of promising antifoams on the chemical precipitation. Based on these evaluations two candidate soaps - Ivory and ML-LL - as well as their companion antifoam agents - DB-110 and SWS-211 - were selected for further work.

Using the two most promising soaps, operating parameters were investigated. These included the effect of soap concentration, ferric chloride concentration, duration of mixing, and pore size of depth filters on the degree of soap removal. The effect of pressure on water flow through filter cartridges and on the rate of decline of water flow was also investigated.

The culmination of the program was the recommendation of a pretreatment concept based on chemical precipitation followed by pressure filtration.

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I. INTRODUCTION

Over the past decade, reverse osmosis has emerged as a convenient and efficient technique for purification of brackish and waste waters. The RO systems are generally compact, the energy requirements are relatively low since the water is not forced through a phase change, and with proper system design it is possible to obtain potable water (i.e., less than 500 ppm dissolved solids) in a single pass.

With increasingly long space flights taking place and with the possibility of orbiting space stations, it has become necessary for NASA to develop techniques to conserve and reclaim water. Perhaps the single greatest source of contaminated water from such missions is wash water from bathing and clothes washing. A typical wash water might contain approximately 0.10 percent detergent or soap and 0.05 percent NaCl; lesser amounts of urea, lactic acid, and phosphate builders; and trace amounts of miscellaneous suspended and colloidal materials such as lint, viruses, bacteria, grease, and soil. It is only natural that NASA is considering membrane separation as a basis for such a development.

Unfortunately, most of the membranes currently available have been designed primarily for salt rejection, and their operational life is adversely affected by wash water components such as detergents, bacteria, soaps, and divalent metal compounds as well as the 165°F pasteurization temperature that is sometimes employed. If the objectionable constituents could be removed by a pretreatment scheme before the wash water passed through the membrane, the membrane's operation and durability would be enhanced.

Under Contract NAS 9-13536, Springborn Laboratories (formerly DeBell & Richardson, Inc.) studied various techniques for eliminating objectionable wash water waste constituents. It was determined by this study that removal of objectionable constituents by a pretreatment scheme is a feasible approach. Laboratory scale tests demonstrated successful pretreatment

schemes for chemical precipitation, filtration, and adsorption. These results were obtained using a simulated wash water containing Olive Leaf Soap.

Under Contract NAS 9-14518, the pretreatment work was expanded to include other soaps such as Ivory and Palmato. In addition, preliminary work was started on selecting antifoam agents for use with the various candidate surfactants.

It was the purpose of the present study to provide for continued development of a wash water pretreatment scheme, resulting in an optimum concept for removing objectionable materials from spacecraft wash water waste prior to its introduction into a reverse osmosis membrane system (currently under development by NASA).

During this study we also selected optimum cleansing and antifoam agents.

II. SELECTION OF ANTIFOAM AGENTS

As part of wash water renovation, it will be necessary to prevent or eliminate the foams that are often encountered with agitated soapy water. One method of eliminating foams is through the use of antifoam agents.

Under Contract NAS 9-14518, we evaluated various candidate antifoams using the "shake test". As a result we were able to identify promising agents for Olive Leaf Soap, Ivory Soap, Neutrogena Rain Bath Gel, Neutrogena Bar Soap, Miranol JEM, Palmeto, and others. In general, the silicones and the heavy metal ions (i.e., ferric, aluminum, etc.) were the most effective antifoams.

In our current work we continued our evaluation with the goal of selecting an optimum antifoam agent for each candidate soap. Antifoam agents which showed promise by the shake test were given a comparative evaluation using a dynamic testing procedure.

As part of this effort, we also evaluated ultrasonic as a defoaming technique.

ULTRASONIC AS A DEFOAMING TECHNIQUE

We conducted a cursory evaluation of ultrasonic for defoaming, using a Sonogen Ultrasonic Cleaner (Model LG 40 - Branson Ultrasonic Corporation), a bath-type unit with a one-liter capacity. The procedure, a modified "shake test", was as follows:

- . 50 cc of the appropriate synthetic wash water was added to a 38 x 200 mm test tube.
- . The tube was then shaken for 15 seconds to generate the foam.
- . The tube was immersed in the ultrasonic bath for 30 seconds.
- . After 15 and 30 seconds the foam height was measured.

Evaluations were done on synthetic wash waters based on the following formulation:

<u>Material</u>	<u>Concentration (ppm)</u>
Surfactant (soap)	1000 ^(a)
Sodium chloride	500
Sodium sulfate	150
Lactic acid	100
Urea	50

(a) Solids or active soap

Table 1 lists the results found with an Ivory Soap and a Palmeto based wash water. Appropriate controls were also run using either no antifoam or a promising silicone. Based on this cursory evaluation, ultrasonic has no promise as a defoaming technique.

TABLE 1

The Effect of Ultrasonic on Foam Height
With
Ivory and Palmeto Based Wash Waters

Defoamer	Foam Height (cm)			
	Palmeto Wash Water		Ivory Wash Water	
	15 sec	30 sec	15 sec	30 sec
Control (no anti-foam)	4.0	3.5	6.5	6.0
	4.6	4.3	7.5	7.0
Silicone (250 ppm SWS 211)	0.1	0.0	-	-
	0.1	0.1	-	-
Ultrasonic	3.6	3.6	7.5	7.2
	5.2	4.7	7.5	7.0

EVALUATION OF ANTIFOAM AGENTS

During the program we continued our preliminary evaluation of antifoams using a new candidate soap, ML-11. This soap from Rochester Germicide is a more concentrated version of Palmeto (39 percent versus 19 percent solids). Due to the similarity to Palmeto, we evaluated only those antifoams which were promising with Palmeto.

Foam heights were determined as before, using a 250 ml graduated cylinder and the NASA "shake test" procedure⁽¹⁾. The results are presented in Table 2. The silicones were generally effective at the 400 ppm level. Ferric chloride was borderline at 150 ppm.

During Contract NAS 9-14518, the silicones as a group demonstrated a high degree of effectiveness in eliminating foam. For this reason we chose to evaluate other silicones during the present program.

Table 3 presents the results of the continued evaluations, using both Ivory and Palmeto based wash waters. For new candidate antifoams used with Palmeto based wash water, results were no better than with agents tried previously, such as DB-110 and SWS-211⁽²⁾.

With Ivory based wash water the new silicones were not as effective as the previously tested DB-110 and AF-71.

DYNAMIC ANTIFOAM STUDIES

In order to conduct dynamic testing, we constructed a continuous recirculating fluid system consisting of a variable-speed "Micro-Pump" centrifugal pump, a 3 inch diameter by 40 inch long glass tube holding tank, and Tygon tubing interconnecting fluid lines. The parts were assembled in such a way that the fluid exited from the bottom of the long cylindrical hold tank, passed through the pump, and then cascaded back into the hold tank. This cascading action generated the foam, which was measured as a steady-state foam height in the tank.

The procedure for testing was as follows:

- . A quantity of wash water plus appropriate antifoam totaling 1900 cc was charged into the glass reservoir.
- . The variable-speed pump, already set to deliver a flow rate of 3 liters per minute, was turned on and the water was allowed to cascade into the glass tube.
- . Since the foam height tends to increase with duration of agitation, even with antifoams, the height was measured after 1, 2, and 5 minutes.

In general, the silicone antifoams were extremely effective in reducing foaming with various types of wash water. Dynamic foam heights, typically, were reduced from 20 to 40 cm down to no more than 1 to 1-1/2 cm at dosages of from 100 to 300 ppm antifoam solids.

Miranol JEM based wash water, which exhibits a foam height of 13 cm after 5 minutes of agitation (Table 4), can be effectively defoamed with 50 ppm of either AF-75 or DB-110. Colloid 680, a proprietary blend, is effective at a dosage of 200 ppm.

Palmeto soap is a high foamer; 5 minutes of agitation with a wash water control produced a foam height of 48 cm (Table 5). Relatively large quantities of silicone antifoam are required to reduce the foam. Antifoams AF-75, SWS-211, and DB-110 are all effective at the 200-300 ppm level.

The ML-11 soap is also a high foamer, producing 43 cm of foam in a 5-minute period (Table 6). An ML-11 based wash water can be defoamed with as little as 100 to 200 ppm of either AF-93, AF-75, or SWS-211. Ferric chloride is also effective, but has the side effect of producing a sludge. In a final pretreatment system, this sludge could foul water-handling components such as centrifugal separators and valves.

Neutrogena based wash water produced surprising results; the control with no antifoam generated only 1 to 1-1/2 cm of foam in 5 minutes of mixing. For this reason, no antifoams were evaluated with this wash water.

As in the case of the other fatty acid soap, Ivory soap is a high foamer, generating 48 cm of foam in 5 minutes of agitation (Table 7). Wash water based on Ivory soap can be effectively defoamed with 200 ppm of any one of the four silicone antifoams evaluated. Ferric chloride is effective, also, but again produces an undesirable soap sludge.

From the above discussion it can be seen that there are a number of anti-foams that are effective in defoaming each of the candidate cleansing agents. Of course the antifoam of choice must also be compatible with the soap removal process.

TABLE 2

Effect of Antifoams on Foam Height⁽¹⁾
With ML-11⁽²⁾ Based Wash Water

Antifoam	Foam Height (cm) at Various Dosages									
	100 ppm		150 ppm		200 ppm		300 ppm		400 ppm	
	15 sec	60 sec	15 sec	60 sec	15 sec	60 sec	15 sec	60 sec	15 sec	60 sec
Ferric Chloride	0.4	0.2	0.2	0.2	2.2	2.0	-	-	-	-
	0.4	0.1	-	-	2.3	2.1	-	-	-	-
AF-75 (3)	3.5	-	-	-	0.3	0.2	0.4	0.2	0.1	0.1
	-	-	-	-	0.7	0.5	0.4	0.2	-	-
SWS-211 (4)	-	-	-	-	0.5	0.2	0.2	0.1	0.1	0.1
	-	-	-	-	-	-	0.2	0.1	0.0	0.0
DB-110 (5)	-	-	-	-	2.0	-	0.9	0.4	0.8	0.1
	-	-	-	-	-	-	1.2	0.4	1.5	-
AF-93 (3)	-	-	-	-	1.0	0.2	0.3	0.0	0.0	0.0
	-	-	-	-	0.9	0.2	0.1	0.1	0.0	0.0
AF-60 (3)	-	-	-	-	-	-	0.4	0.2	0.3	0.1
	-	-	-	-	-	-	-	-	0.4	0.1

(1) NASA "shake test"

(2) Rochester Germicide

(3) General Electric Company

(4) SWS Silicones; Division of Stauffer Chemical Company

(5) Dow Corning Corporation